# ermal Analysis Excellence



#### DSC 3+

2000	
STAR <sup>e</sup> System	
Innovative Technology	
Versatile Modularity	
Swiss Quality	



# Differential Scanning Calorimetry For Unmatched Performance



# **Unmatched DSC Performance** Tailored Exactly to Your Needs

Differential scanning calorimetry (DSC) is the most frequently used thermal analysis technique. DSC measures enthalpy changes in samples due to changes in their physical and chemical properties as a function of temperature or time.

Features and benefits of the METTLER TOLEDO DSC 3+:

- Amazing sensitivity for the measurement of weak effects
- Outstanding resolution allows measurement of rapid changes and close-lying effects
- Robust endurance-tested sample robot operates efficiently and reliably around the clock
- Small and large sample volumes for microgram or inhomogeneous samples
- Modular concept tailor-made solutions for current and future needs
- Flexible calibration and adjustment guarantees accurate results under all conditions
- Wide temperature range from -150 to 700 °C in one measurement





The DSC utilizes innovative DSC sensors with 56 (FRS) or 120 (HSS) thermocouples which guarantee unbeatable sensitivity and outstanding resolution.

#### www.mt.com/ta-dsc

# **Breakthrough in DSC Sensor Technology** Unsurpassed Sensitivity and Resolution

Don't make any compromises concerning the sensor, the heart of your DSC. The METTLER TOLEDO MultiSTAR<sup>™</sup> sensors successfully combine a number of important characteristics that are unattainable with conventional sensors and that until now have been impossible to achieve. These included high sensitivity, excellent temperature resolution, a perfectly flat baseline and robustness.

# Temperature resolution





The signal time constant determines how well close-lying or overlapping thermal effects are separated from one another. Our ceramic sensor material provides excellent performance due its low thermal mass and high thermal conductivity.

# Non Benefine DSC 3+ FIRS 6-- Vasit converted Intelligence DSC 3+ / FIRS 6-- sensor DSC 3+ / FIRS 6-- sensor Blank convected 0 Blank convected

Our revolutionary star-shaped arrangement of thermocouples around the sample and reference crucibles completely compensates any possible temperature gradients. This guarantees flat baselines and reproducible measurement results.

# Sensitivity

A quantum jump in sensor technology enables us to offer the highest sensitivity sensors available in DSC instrumentation and allows you to detect the weakest thermal effects. The signal-tonoise ratio, an important instrument parameter, is determined by the number of thermocouples and their specific arrangement.



Measurement principles: Differential scanning calorimetry (DSC) measures the difference between the heat flows from the sample and reference sides of a sensor as a function of temperature or time.

# **DSC 3+ from METTLER TOLEDO** The Right Decision

The DSC 3<sup>+</sup> has a modular design, making it an ideal choice for automatic or manual operation, from industrial development and academic research to production and quality assurance.





If the DSC is not installed next to a PC running the STAR<sup>e</sup> software, you can set up individual sequences directly at the instrument terminal. The adaptable and intuitive touchscreen or SmartSens allows you to switch screens or open the furnace handsfree.

#### OneClick™ function



The terminal with the OneClick function is clearly visible even at a distance and provides information on the status of the measurement. The OneClick function allows you to easily and efficiently start a predefined method.

#### Ergonomics in perfection



The ergonomic design has been improved: If you insert samples manually, you can rest your hand on an ergonomically shaped support surface.



#### **Two-level calibration offering**

#### **Standard Calibration**

Standard Calibrations on any thermal analysis instrument support one heating rate

- Procedures are carried out according to GMP-approved Standard Operational Procedures (SOPs)
- Automatically generates METTLER TOLEDO standardized certificates



#### **Expert Calibration**

 $\label{eq:expert} \mbox{Expert Calibration} - \mbox{ideal for DSC and TGA/DSC} - \mbox{supports multiple} \\ \mbox{heating rates}$ 

- Procedures are carried out according to GMP-approved Standard Operational Procedures (SOPs)
- Automatically generates METTLER TOLEDO standardized certificates



Calibration is key at METTLER TOLEDO. Benefit from our unique calibration and adjustment services for your thermal analysis instrumentation:

- Professional adjustment by supplier-trained experts improves result accuracy
- Supplier certificates in accordance with regulatory requirements
- www.mt.com/ta-calibration

# **Reliable Automation** 24 Hours a Day, Like a Swiss Watch

Automatic and efficient, our sample robot is a sophisticated automation option allowing for reliable operation 24 hours a day though the whole year. Together with the STAR<sup>e</sup> software, the power of the automation is increased by our unique FlexCal<sup>™</sup> calibration concept always selecting the correct adjustment parameters, and the possibility to automatically evaluate results.

## Unique lid piercing accessory



The sample robot can remove the protective crucible lid from the crucible or can pierce the lid of hermetically sealed aluminum crucibles immediately before measurement. This unique feature prevents the sample from taking up or losing moisture between weighing-in and measurement. It also protects oxygen-sensitive samples from oxidation.

#### Automatic furnace lid



The automatic furnace lid opens and closes the furnace chamber at a keystroke or when actuated by the SmartSens infrared sensors. Manual removal and replacement of the furnace lid is no longer necessary. The measurement cell is effectively isolated from the environment thanks to its optimized design with three superimposed silver lids and its heat shield. Universal gripper



The universal gripper can handle all types of METTLER TOLEDO crucibles.



All our DSC models can be automated. The sample robot can process up to 34 samples even if every sample requires a different method or a distinct crucible.

# Innovative Accessories Increases Measurement Power

A DSC instrument can be easily equipped with an optical accessory, such as a microscope or photocalorimeter. DSC microscopy allows the user to capture images of the sample while it is heated or cooled in the DSC. DSC Photocalorimetry exposes a sample to light of a particular wavelength range and intensity for a defined time and records the heat flow from the sample.

#### DSC microscopy



DSC curves often exhibit effects that cannot immediately be explained. In such cases, it is often helpful to visualize the changes in the sample directly by means of microscopy. The versatile optical accessory can be used with any METTLER TOLEDO DSC. It consists of an optical system, a CCD camera, and image capture and processing software.

#### www.mt.com/ta-optics

#### **DSC** photocalorimetry



The photocalorimetry accessory for the DSC allows you to characterize UV curing systems. You can study photoinduced curing reactions and measure the effects of exposure time, UV light intensity and temperature on material properties.

#### Light sources for UV measurements



Different types of light sources:

- Light source with a wavelength range (in the UV light)
- Light source with a wavelength range (in the VIS light)
- Light sources based on LED technology (provided wavelengths: 365, or 385 or 400 nm)

Option $\rightarrow$ required option	FRS 6+	HSS 9+	Automatic furnace lid	SmartSens terminal	Peripheral control	Switched line socket	GC 402	Air cooling	Cryostat	Intra- Cooler	Liquid nitrogen
DSC 3+ (500 °C)	•	•	optional	optional				•	•	•	•
DSC 3+ (700 °C)	•	•	optional	optional				•	•	•	•
Sample changer (34)			essential	essential							
Automatic furnace lid				essential							
Gas controller (GC 302)				recom.			optional				
Cryostat/IntraCooler						optional (recom.)					
Liquid nitrogen cooling					essential						

Option Matrix: A DSC 3+ for every need and configuration can be found in the below table.

• = Selectable

Upgrading your system is possible in the future. The modularity of the system lets you buy today and improve it tomorrow. Adding practical accessories or changing the furnace is possible at any time.



# Defined furnace atmosphere, programmable gas flow and gas switching

The furnace chamber can be purged with a defined gas flow. The software-controlled mass flow gas controller measure and regulates the gas flow between 0 and 200 mL/min and can automiatically switch up to 4 gases. Regulate and switch gases such as air, nitrogen, oxygen, argon,  $CO_2$  and inert hydrogen (96% Ar, 4% H<sub>2</sub>).

Air cooling	RT to 500 °C / 700 °C
Cryostat cooling	–50 to 450 °C / 700 °C
IntraCoolers (several)	−35 to 450 °C / 700 °C −85 to 450 °C / 700 °C −100 to 450 °C / 550 °C
Liquid nitrogen cooling	–150 to 500 °C / 700 °C

#### Temperature range and cooling options

You can adapt the system to your requirements depending on the temperature range in which you want to measure. The IntraCooler is a sealed system requiring only electrical power. It is therefore advantageous in locations where liquid nitrogen is undesirable or not available. Liquid nitrogen cooling offers greater flexibility because it allows you to measure over the entire temperature range.

# **Crucibles for Thermal Analysis** Guarantee Reliable Results

Crucibles serve as containers for samples during thermoanalytical measurements. They guarantee that the sensor is not contaminated by the measurement. The type of crucible used for a measurement can have a large effect on the quality of the results obtained, and in addition, also influences important characteristics of the DSC measuring cell. Considering the relevant factors before the measurement can often help to save time later on when interpreting the curve.



We have the right crucible for every application. The crucibles are made of different materials with volumes ranging from 20 to  $160 \ \mu L^*$  (900  $\mu L$  all techniques) and for high pressures. All the different types can be used with the sample robot. Available crucibles can be found here:

www.mt.com/ta-crucibles

#### \* with furnace expander

Crucible sealing press and sealing tools



The press allows the pan to be sealed very easily. Under the pressure of the plunger the pan is cold welded, hermetically sealing with the lid. After changing plunger and die you can use the press for other crucibles.

Crucible handling set



The crucible handling set provides a range of tools that are fundamental for sample loading and pan and lid handling:

- A funnel for filling the pan with sample
- Tweezers for handling of sample, pans and lids
- Different type of needles and a piece of rubber for lid preparation
- Crucible holder for crucible handling and safe bringing to the instrument



- 1. Furnace lid
- 2. Crucibles on the DSC sensor
- 3. Silver furnace
- 4. PT100 of furnace
- 5. Flat heater between two insulating disks
- 6. Thermal resistance for cooler
- 7. Cooling flange
- 8. Compression spring construction
- 9. Cooling flange
- 10. DSC raw signal for amplifier
- 11. Purge gas inlet
- 12. Dry gas inlet

Differences in heat flow arise when a sample absorbs or releases heat due to thermal effects such as melting, crystallization, chemical reactions, polymorphic transitions, vaporization and many other processes. Specific heat capacities and changes in heat capacity, for example during a glass transition, can also be determined from the difference in heat flow.

# **Extremely Wide Application Range** For All Kind of Materials

Differential scanning calorimetry measures the enthalpies associated with transitions and reactions and the temperatures at which these processes occur. The method is used for the identification and characterization of materials.

Differential scanning calorimetry (DSC) is fast and very sensitive. Sample preparation is easy and requires only small amounts of material. The technique is ideal for quality control, material development and material research.

#### Examples of thermal events and processes that can be determined by DSC

Melting behavior

- Curing
- Crystallization and nucleation
- ad Polymorphism
- Liquid-crystalline transitions
- Phase diagrams and composition
- Glass transitions
- Reactivity
- Reaction kinetics

- Stability
- Miscibility
- Effects of plasticizers
- Thermal history
- Heat capacity and heat capacity changes
- Reaction and transition enthalpies
- Purity





The method is used to analyze and study materials such as thermoplastics, thermosets, elastomers, composite materials, metals and alloys, adhesives, foodstuffs, pharmaceuticals and chemicals.

#### www.mt.com/ta-applications

#### Influence of accelerator on curing



Curing measurements of samples of glass-fiber-reinforced vinyl ester resin composites containing different concentrations of an accelerator can be used to determine the influence of a particular accelerator on the curing process. Each DSC curve exhibits an exothermic peak proportional to the heat produced during the exothermic curing reaction. Increased accelerator concentration speeds up the reaction and causes the reaction peak to shift to lower temperatures. This type of DSC measurement allows production processes to be adapted and optimized by choosing the right accelerator, its concentration, and the curing temperature.

#### Liquid crystals



Materials consisting of relatively stiff molecules can form liquid-crystal phases. This behavior is demonstrated in the example showing DSC measurements of LC (R) MHPOBC. The substance exhibits several liquid-crystal transitions above the melting temperature at 85 °C. The transitions that occur between 114 and 124 °C are very weak and are displayed in the zoomed region of the cooling curve. Since liquid-crystal transitions often produce only very small thermal effects, the DSC used to measure such transitions must have high resolution and low noise performance specifications.

#### Polymorphism



The analysis of melting behavior is an important method for the quality control of pharmaceutical products. As the blue curve in the example shows, the melting curve of the stable form of phenobarbital can be used to determine the melting point and for purity determination. DSC is also used to study polymorphic forms. The red curve shows that the metastable form first melts at a lower temperature. The melt then crystallizes to the stable form before this form also melts. Knowledge of the particular crystalline form present is very important for assessing the physical stability of substances.

#### Nanocrystallization



The crystallization behavior of an amorphous metal alloy prepared by rapid cooling in a meltspinning production process was analyzed. The diagram displays curves measured at different heating rates. The exothermic peak from 470 °C onward is due to the formation of the iron-silicon nanocrystallites. With increasing heating rates, the maximum of this peak shifts to higher temperatures. The relationship between the crystallization temperature and the heating rate yields information on the activation energy of the crystallization process. The highly asymmetric shape of the peak reveals additional information about the kinetics of crystallization.

#### O/W cream formulations



Creams are semi-solid emulsions and consist of mixtures of oil and water. Differences in the content of constituents can affect the consistency and quality of the finished cream formulation. DSC curves of two different creams exhibit a large melting peak in the range 55 to 65 °C. This peak is due to the presence of glyceryl monostearate, which acts as a thickening agent and stabilizer. Cream A, however, has three additional peaks in the region 25 to 45 °C, which are the typical melting points of mono-, di- and tri-glycerides. These inactive ingredients form three-dimensional structures of different types and strengths.



Information about the stability of materials can be obtained from the analysis of decomposition reactions. One widely used standard test method is the measurement of the oxidation induction time, OIT. This is the time up to the onset of oxidation when a sample is held isothermally at a certain temperature in an oxygen atmosphere. In the example, the OITs of three polyethylene samples stabilized to different extents were measured at 210 °C. The differences in stability toward oxidation can be clearly seen. These measurements also allow thermally, mechanically or chemically stressed material to be distinguished from fresh material.

#### **Oxidation stability**

#### **Curing of adhesives**



When an adhesive cures isothermally, the material changes from a liquid to a solid as a result of a chemical reaction. An amorphous polymeric glass is formed and the reaction practically stops. This process is known as vitrification and is of great practical importance because a vitrified adhesive is not fully cured and is therefore unstable. The properties of the material gradually change over a long period of time. The example shows that measurement of the heat capacity during the curing reaction using **TOP**EM<sup>™</sup> (a temperature-modulated DSC technique) is a simple and reliable method to identify vitrification processes.

#### **Vulcanization reaction**





DSC results can be used to analyze and predict the kinetics of vulcanization reactions using Model Free Kinetics (MFK) software. In Step 1, three dynamic DSC curves of acrylonitrile-butadiene rubber (NBR) were measured at different heating rates. The results were then used to calculate the conversion curves shown in Step 2. From this data the activation energy curve of the reaction was calculated as a function of conversion as shown in Step 3. The activation energy curve was then used to predict the isothermal kinetics at 130 °C. (green curve in Step 4). The prediction is in excellent agreement with data measured under the same conditions shown by the solid black squares.

# Simple, Intuitive Operation Straightforward, Efficient and Secure

STAR<sup>e</sup> software has been expanded to include new features that help you prepare your DSC 3+ instrument for specific experiments, develop methods for advanced analyses and perform flexible result evaluations. Complex measurement programs are set up within minutes and the vast range of available tools permit curves to be evaluated both accurately and efficiently.

### Asset management



#### TOPEM<sup>™</sup>



Obtain a detailed overview of a thermal analysis system, even if installed in multiple laboratories or sites. Monitor each instrument, including its date of purchase, calibration history and service log. Data can also be exported in CSV format for upload to an existing company-wide asset management system.

The methods for determining  $c_p$ are described in various standards including, ISO 11357-4, DIN 51007 and ASTM E1269. S**TA**R<sup>e</sup> software supports the following methods to determine  $c_p$  from the DSC heat flow curve:

- 1. Direct
- 2. Sapphire (DIN)
- 3. IsoStep
- 4. Steady State
- 5. ADSC
- 6. **TOP**EM

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www.mt.com/ta-cp
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#### Analyses the Competition Total International DSC Conversional DS

TMDSC methods allow both temperature-dependent and time-dependent processes to be separated. The basic idea of **TOP**EM is to overlay the isothermal or ramped temperature with a time series of stochastic (random) temperature pulses of different durations.

www.mt.com/ta-topem

# Complete Thermal Analysis System



A complete thermal analysis system consists of the basic six complementary measuring techniques, each of which brings fast and accurate results. Additional knowledge can be obtained by means of several hyphenated techniques.

#### www.mt.com/ta-software

# World-Class Service and Support Provide Results You Can Trust

METTLER TOLEDO's portfolio of services is designed to ensure the continuous performance and reliability of your thermal analysis systems. Factory-trained in Switzerland, our worldwide teams bring the professional expertise and know-how needed to provide you with the highest level of after-sales support, as well as the experience necessary to optimize services for your own particular needs.





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# Bi-annual application magazine



Every year, thermal analysis generates a large number of scientific results and discoveries. Interesting examples from different application fields and industries are published in our UserCom magazine.

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#### **Differential Scanning Calorimetry**

nal Analysis Premium

Differential Scanning Calorimetry (DSC) allows you to determine the energy absorbed or released by a sample as it is heated or cooled.



A section through a DSC measuring cell

- 1 Fumore lid
- 2 Duritie on Ne DEC Service
- 3 Sher turnese
- 4 PTOD temperature sensor of fummos
- 5 Fot hader tabaset has insubling its 6 Thermal excitance for cooler
- 7 Castling Range
- 8 Compression spring or
- 8 Cooling Range P100
- 10 DSC now signal for smplitter
- 11. Purge gas intel
- 12 Dry gas mer

#### Features and Benefits of the DSC

- Amazing samellisity for the mecourement of week effects
- Datatending resolution ottows measurement of repid stranges and stone-lying affects
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Learn more about thermal analysis and its applications for different industries by making use of our comprehensive collection of live and recorded webinars. Presented by thermal analysis experts, you will also be given the opportunity to ask questions at the end of each seminar.

www.mt.com/ta-webinars

#### **DSC 3+ Specifications**

#### Temperature data

air cooling	RT to 500 °C (200 W)	RT to 700 °C (400 W)		
cryostat cooling	–50 to 450 °C	–50 to 700 °C		
IntraCooler	-100 to 450 °C	-100 to 700 °C		
liquid nitrogen cooling -150 to 500 °C		-150 to 700 °C		
	± 0.2 K			
Temperature precision <sup>1)</sup>		± 0.02 K		
Furnace temperature resolution		± 0.00006 K		
Heating rate <sup>2)</sup> RT to 700 °C		0.02 to 300 K/min		
Cooling rate <sup>2)</sup>		0.02 to 50 K/min		
air cooling	8 min (500 to 100 °C)	9 min (700 to 100 °C)		
cryostat cooling	5 min (100 to 0 °C)			
IntraCooler	5 min (100 to 0 °C)			
liquid nitrogen cooling	15 min (100	) to -100 °C)		
	FRS 6+	HSS 9+		
Sensor material		Ceramic		
Number of thermocouples		120		
Signal time constant		3.1 s		
raw data	19.5 <sup>3)</sup>	6.9		
mathematically corrected	> 155 4)	> 85 4)		
resolution	0.12	0.20		
sensitivity	11.9	56.0		
at 100 °C	± 350 mW	± 160 mW		
at 700 °C	± 200 mW	± 140 mW		
Resolution		0.02 µW		
Digital resolution		16.8 million points		
Sampling rate		maximum 50 values/second		
	air cooling cryostat cooling IntraCooler liquid nitrogen cooling on air cooling cryostat cooling IntraCooler liquid nitrogen cooling raw data mathematically corrected resolution sensitivity at 100 °C at 700 °C	air cooling         RT to 500 °C (200 W)           cryostat cooling         -50 to 450 °C           IntraCooler         -100 to 450 °C           liquid nitrogen cooling         -150 to 500 °C           iquid nitrogen cooling         -150 to 500 °C           iquid nitrogen cooling         -150 to 500 °C           iquid nitrogen cooling         ± 0.           con         ± 0.000           on         ± 0.000           con         ± 0.000 °C           air cooling         8 min (500 to 100 °C)           cryostat cooling         5 min (100           IntraCooler         5 min (100           IntraCooler         5 min (100           Iiquid nitrogen cooling         15 min (100           raw data         19.5 ³)           mathematically corrected         > 155 <sup>4</sup> )           resolution         0.12           sensitivity         11.9           at 100 °C         ± 350 mW           at 700 °C         ± 200 mW           0.04 µW         16.8 mill		

#### **Special modes**

•			
ADSC	standard		
IsoStep <sup>™</sup> , <b>TOP</b> EM <sup>™</sup>			
Automation	ontional		
Microscopy	opiionai		
Photocalorimetry			

#### Approvals

IEC/EN61010-1:2001, IEC/EN61010-2-010:2003 CAN/CSA C22.2 No. 61010-1-04 UL Std No. 61010A-1 EN61326-1:2006 (class B) EN61326-1:2006 (Industrial environments) FCC, Part 15, class A AS/NZS CISPR 22, AS/NZS 61000.4.3 Conformity mark: CE

Specifications and approvals for the IntraCooler option are only valid for systems with Huber coolers.

1) based on metal standards

<sup>2)</sup> depends on instrument configuration

<sup>3)</sup> no mathematical treatment to the data or correction applied <sup>4)</sup> corrected according to B. Wunderlich, Thermal Analysis of

Polymeric Materials, Springer (2005), page 346

#### www.mt.com/ta,

For more Information



Quality certificate. Development, production and testing according to ISO 9001.



Environmental management system according to ISO 14001.

**C C European conformity**". The CE conformity mark provides you with the assurance that our products comply with the EU directives.

#### **METTLER TOLEDO Group**

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